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COMPLETED PROJECT SUMMARY

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PRINCIPAL INVESTIGATOR: Professor Thomas Engel
Department of Chemistry
University of Washington
Seattle, WA 98195

INCLUSIVE DATES: 1 April 1987 - 15 February 1991

GRANT NUMBER: AFOSR-87-0166

COSTS AND FY SOURCE: \$120,086 ('87-'88); \$123,550 ('88-'89);
\$125,878 ('89-'90).

SENIOR RESEARCH PERSONNEL: Professor Thomas Engel
Dr. Mark D'Evelyn
Dr. James Engstrom
Dr. Yongkui Sun

JUNIOR RESEARCH PERSONNEL: Mark Nelson
Douglas Bonser

PUBLICATIONS:

1. *Molecular Beam Studies of the Reaction of Silicon with Oxygen*, M.P.D'Evelyn, M. M. Nelson and T. Engel, *J. Vac. Sci. Technol. A5* (1987), 642.
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The following objectives were set at the outset of the grant period.

- (1) To measure the absolute adsorption probabilities of O_2 , O, F_2 and F and to compare the atomic and molecular reactivities.
- (2) To measure the structural variation of the adsorption probability by comparing results on Si(100) and Si(111).
- (3) To determine the reaction mechanisms for the reactions listed above.
- (4) To determine the kinetic parameters (activation energy and preexponential factor) for these reactions.
- (5) To determine the intermediates in these reactions by carrying out time resolved XPS of the chemically shifted atomic levels.

All but the last objective have been studied in some detail and many issues have been clarified. Atomic oxygen is much more reactive than O_2 at all temperatures and coverages studied. Atomic fluorine is much more reactive at high coverages but is only slightly more reactive than F_2 at low coverages. The kinetics for the reaction $Si + F_2 \rightarrow SiF_2$ and $Si + O_2 \rightarrow SiO$ have been studied in detail. The study of surface intermediates using XPS has not been successful because the chemical shifts for different intermediates were not sufficiently large to resolve using a laboratory X-ray source. We will attempt to obtain the same information using time resolved SIMS.

Final Technical Report

Grant No. AFOSR-87-0166

Principal Investigator: Professor Thomas Engel

Period: April 1987 - February 1991

Completed work

1. The Reaction of Fluorine with the Si(100) Surface

The kinetics of the thermal decomposition of silicon-fluoride adlayers have been examined under ultrahigh vacuum conditions with temperature-programmed desorption, modulated molecular beam techniques and steady-state reaction measurements employing both X-ray photoelectron spectroscopy (XPS) and mass spectrometry. Temperature-programmed decomposition of silicon-fluoride adlayers, produced by exposing the clean Si(100) surface at 120 K to a beam of fluorine, yielded $\text{SiF}_2(\text{g})$ and $\text{SiF}_4(\text{g})$ as the only gas phase reaction products. The relative yield of these two gas phase reaction products depends strongly on the initial coverage of fluorine adatoms — below approximately 3 ML, $\text{SiF}_2(\text{g})$ is the major reaction product, whereas above approximately 3 ML, the yield of $\text{SiF}_2(\text{g})$ remains constant while that of $\text{SiF}_4(\text{g})$ increases continuously. Above initial coverages of 2 ML, the thermal decomposition is terminated near 800 K by the removal of one monolayer of the silicon substrate in the form of $\text{SiF}_2(\text{g})$. A detailed analysis of the decomposition reaction for coverages below 2 ML revealed zero-order kinetics spanning the range of 1.0 to 0.3 ML. A qualitative assessment of the adlayer configuration following partial decomposition suggests that the thermal decomposition in the zero-order regime proceeds inhomogeneously, leaving separate domains where the *local* coverage of fluorine is either near saturation or zero. We suggest that the spatially inhomogeneous decomposition is a manifestation of preferential reactivity at surface defects such as atomic steps. In the limit of very low fluorine adatom coverages, modulated molecular beam measurements indicate that the reaction that forms $\text{SiF}_2(\text{g})$ follows second-order kinetics. We suggest that the second-order kinetics is a manifestation of a pseudo-equilibrium between $\text{SiF}(\text{a})$ and $\text{SiF}_2(\text{a})$ species, with the decomposition reaction limited by desorption of $\text{SiF}_2(\text{a})$. Investigation of the steady-state reaction between $\text{F}_2(\text{g})$ and the Si(100) substrate for temperatures of 650-1200 K indicates

conclusively that fluorine must be adsorbed dissociatively for the gasification reaction [under these conditions, production of $\text{SiF}_2(\text{g})$] to occur, i.e., a Langmuir-Hinshelwood mechanism dominates.

2. The Adsorption and Reaction of Atomic Oxygen with the Si(100) Surface

The interaction of atomic oxygen with the Si(100) surface has been examined under UHV conditions with X-ray photoelectron spectroscopy, mass spectrometry, and ion scattering spectroscopy. The supersonic beam of oxygen atoms is produced by employing a high pressure (≈ 100 Torr) RF glow discharge. Both the adsorption and incorporation of oxygen atoms into the Si lattice have been examined as functions of the substrate temperature, the average beam energy and the angle of incidence of the oxygen atoms. The initial probability of adsorption of $\text{O}(\text{g})$ on the Si(100) surface is equal to 1.0 ± 0.1 . This value is essentially independent of both substrate temperature ($T_s = 120\text{--}800$ K) and average beam energy ($\langle E_{\text{av}} \rangle = 4\text{--}20$ kcal-mol $^{-1}$). The adsorption probability decreases monotonically with increasing coverages of oxygen and is characterized by two regimes — a rapid adsorption phase persists to approximately 3 ML and is characterized by a near unity probability of adsorption, whereas for coverages above approximately 5 ML, the adsorption probability is considerably reduced and is approximately equal to 10^{-2} . At sufficiently high temperatures ($T \geq 900$ K) the adsorption of oxygen atoms leads to the gasification of the Si substrate via the reaction $\text{O}(\text{g}) + \text{Si}(\text{s}) \rightarrow \text{SiO}(\text{g})$. Analysis of this reaction via modulated beam techniques implicates first-order kinetics. This result can be compared with that obtained for molecular oxygen where a sequential, two-step first-order process was found to apply. Moreover, the kinetic parameters for the reaction involving $\text{O}(\text{g})$ are *identical* to those found for the slow reaction step involving $\text{O}_2(\text{g})$. We interpret this finding as evidence for direct attachment of oxygen atoms to the dangling bond surface states on the Si(100) surface, the resulting "silicon monoxide" species desorbing via a first-order reaction.

We have found that the rates for SiO production from oxygen covered Si(100) and Si(111) depend strongly on the oxygen coverage and residence time of oxygen. In modulated beam experiments in which the coverages are below 10^{-2} monolayers and the residence time of the adatoms is less than 10^{-3} seconds, the rate constant for SiO desorptions is 100-1000 times

greater than in conventional thermal desorption experiments. In these experiments, coverages are generally 0.1-1.0 monolayers and the residence time is greater than 10 seconds. In a recently submitted paper, we propose two kinetic models to explain this result. They suggest that only certain surface intermediates are accessed in each of the two techniques.

3. The Effect of Coincident Ion Bombardment on the Oxidation of Si(100) by Atomic Oxygen.

We have measured the rate of oxidation of Si(100) by atomic oxygen with and without an incident Ar^+ beam for substrate temperatures of 300K and 850K. Since the rate of oxygen buildup in the absence of the beam, the rate of oxygen sputtering through the Ar^+ beam and the overall rate of oxidation with both beams incident on the surface can all be measured independently using XPS, we can separate the overall processes into elementary steps. We find that the rate of ion induced adsorption to thermal desorption can be greater than a factor of 10 above 7 monolayers coverage. The removal of adsorbed oxygen by sputtering reduces the ratio of the ion induced rate minus the sputtering rate to the thermal rate to about 3. We also find that the ion induced adsorption at 300K leads to a substantial amount of suboxides relative to the adsorption layer formed by thermal oxidation. This difference disappears if the process is carried out at 850K, presumably due to enhanced diffusion at the elevated temperature.

4. Inhomogeneous Decomposition of Oxide Layers on Si(100).

Most surface science investigations have great sensitivity normal to the surface, but very little sensitivity lateral to the surface. Therefore, it is generally assumed that thermal decomposition of adlayers proceeds homogeneously with random vacancies. We have shown that this is not the case for SiO desorption from Si(100). We find that oxide layers of thickness as low as one monolayer decompose by void formation. The activation energy and preexponential factors have been determined. We also have found that decomposition occurs by enlargement of the voids without a thinning or a coverage change in those areas which are outside of the voids.

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SUPPORT PERSONNEL:

Postdoctoral Research Associates: Mark D'Evelyn, James R. Engstrom, Yongkui Sun

Graduate Research Assistants: Mark Nelson, Douglas Bonser

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